



# Analytical Laboratory

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13339 Hagers Ferry Road  
Huntersville, NC 28078-7929  
McGuire Nuclear Complex - MG03A2  
Phone: 980-875-5245 Fax: 980-875-4349

## Order Summary Report

**Order Number:** J13110158

Project Name:

Customer Name(s): Bill Kennedy, Wayne Chapman

Customer Address: 3195 Pine Hall Rd  
Mailcode: Belews Steam Station  
Belews Creek, NC 28012

Lab Contact: Jason C Perkins Phone: 980-875-5348

**Report Authorized By:** \_\_\_\_\_ **Date:** 12/3/2013  
(Signature) Jason C Perkins

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### Program Comments:

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

### Data Flags & Calculations:

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted. Subcontracted data included on the Duke Certificate of Analysis is to be used as information only. Certified vendor results can be found in the subcontracted lab final report. Duke Energy Analytical Laboratory subcontracts analyses to other vendor laboratories that have been qualified by Duke Energy to perform these analyses except where noted.

### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

*Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)*

### Certification:

The Analytical Laboratory holds the following State Certifications : North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID's & Descriptions:

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2013027419	BELEWS	06-Nov-13 8:05 AM	TO	FGD Purge Eff
2013027420	BELEWS	06-Nov-13 8:10 AM	TO	EQ Tank Eff
2013027421	BELEWS	06-Nov-13 8:15 AM	TO	BIOREACTOR 1 INF
2013027422	BELEWS	06-Nov-13 8:17 AM	TO	BioReactor 2 Inf
2013027423	BELEWS	06-Nov-13 8:17 AM	TO	BioReactor 2 Eff
2013027424	BELEWS	06-Nov-13 2:15 PM	RG	Bio Pilot Eff
2013027425	BELEWS			FILTER BLANK
2013027426	BELEWS	28-Oct-13 4:35 PM	DB	METALS TRIP BLANK
8 Total Samples				

## Technical Validation Review

### Checklist:

- |  |   |  |
|--|---|--|
| COC and .pdf report are in agreement with sample totals and analyses (compliance programs and procedures). | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No            |
| All Results are less than the laboratory reporting limits.   | <input type="checkbox"/> Yes            | <input checked="" type="checkbox"/> No |
| All laboratory QA/QC requirements are acceptable.  | <input checked="" type="checkbox"/> Yes | <input type="checkbox"/> No            |

### Report Sections Included:

- |   |  |
|---|--|
| <input checked="" type="checkbox"/> Job Summary Report                            | <input checked="" type="checkbox"/> Sub-contracted Laboratory Results                |
| <input checked="" type="checkbox"/> Sample Identification                         | <input type="checkbox"/> Customer Specific Data Sheets, Reports, & Documentation     |
| <input checked="" type="checkbox"/> Technical Validation of Data Package          | <input type="checkbox"/> Customer Database Entries                                   |
| <input checked="" type="checkbox"/> Analytical Laboratory Certificate of Analysis | <input checked="" type="checkbox"/> Chain of Custody                                 |
| <input type="checkbox"/> Analytical Laboratory QC Report                          | <input checked="" type="checkbox"/> Electronic Data Deliverable (EDD) Sent Separatel |

Reviewed By: DBA Account

Date: 12/3/2013

# Certificate of Laboratory Analysis

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*This report shall not be reproduced, except in full.***Order # J13110158**

Site: FGD Purge Eff

Collection Date: 06-Nov-13 8:05 AM

**Sample #: 2013027419**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>INORGANIC IONS BY IC</u></b>								
Bromide	99	mg/L		5	50	EPA 300.0	11/11/2013 15:23	JAHERMA
Chloride	7000	mg/L		100	1000	EPA 300.0	11/11/2013 15:23	JAHERMA
<b><u>MERCURY (COLD VAPOR) IN WATER</u></b>								
Mercury (Hg)	219	ug/L		5	100	EPA 245.1	11/23/2013 10:13	DKJOHN2
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	210	mg/L		0.5	10	EPA 200.7	11/13/2013 12:22	MHH7131
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	147	ug/L		10	10	EPA 200.8	11/20/2013 14:02	DJSULL1
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	172	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Chromium (Cr)	311	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Copper (Cu)	127	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Nickel (Ni)	243	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Selenium (Se)	4080	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Thallium (Tl)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Vanadium (V)	192	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
Zinc (Zn)	246	ug/L		10	10	EPA 200.8	11/18/2013 14:49	DJSULL1
<b><u>SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	Complete					Vendor Method	V_AS&C	
<b><u>TOTAL DISSOLVED SOLIDS</u></b>								
TDS	19000	mg/L		250	1	SM2540C	11/19/2013 14:19	DSBAKE1
<b><u>TOTAL SUSPENDED SOLIDS</u></b>								
TSS	3200	mg/L		250	1	SM2540D	11/12/2013 12:27	DSBAKE1

Site: EQ Tank Eff

Collection Date: 06-Nov-13 8:10 AM

**Sample #: 2013027420**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>MERCURY (COLD VAPOR) IN WATER</u></b>								
Mercury (Hg)	152	ug/L		2.5	50	EPA 245.1	11/23/2013 10:15	DKJOHN2
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	194	mg/L		0.5	10	EPA 200.7	11/13/2013 12:26	MHH7131

# Certificate of Laboratory Analysis

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*This report shall not be reproduced, except in full.***Order # J13110158**

Site: EQ Tank Eff

Collection Date: 06-Nov-13 8:10 AM

**Sample #: 2013027420**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	122	ug/L		10	10	EPA 200.8	11/20/2013 14:05	DJSULL1
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	145	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Chromium (Cr)	264	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Copper (Cu)	105	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Nickel (Ni)	202	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Selenium (Se)	3300	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Thallium (Tl)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Vanadium (V)	177	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1
Zinc (Zn)	197	ug/L		10	10	EPA 200.8	11/18/2013 14:53	DJSULL1

Site: BIOREACTOR 1 INF

Collection Date: 06-Nov-13 8:15 AM

**Sample #: 2013027421**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>NITRITE + NITRATE (COLORIMETRIC)</u></b>								
Nitrite + Nitrate (Colorimetric)	< 0.01	mg-N/L		0.01	1	EPA 353.2	11/15/2013 11:37	BGN9034
<b><u>INORGANIC IONS BY IC</u></b>								
Bromide	86	mg/L		5	50	EPA 300.0	11/11/2013 15:42	JAHERMA
Chloride	6400	mg/L		100	1000	EPA 300.0	11/11/2013 15:42	JAHERMA
<b><u>Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	183	mg/L		0.5	10	EPA 200.7	11/13/2013 12:30	MHH7131
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	88.1	ug/L		10	10	EPA 200.8	11/20/2013 14:09	DJSULL1

# Certificate of Laboratory Analysis

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*This report shall not be reproduced, except in full.***Order # J13110158**

Site: BIOREACTOR 1 INF

Collection Date: 06-Nov-13 8:15 AM

**Sample #: 2013027421**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Nickel (Ni)	24.6	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Selenium (Se)	76.8	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Thallium (Tl)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Vanadium (V)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 14:56	DJSULL1

**SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)**

Vendor Parameter	Complete	Vendor Method	V_AS&C
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**TOTAL DISSOLVED SOLIDS**

TDS	14000	mg/L	25	1	SM2540C	11/19/2013 14:19	DSBAKE1
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**TOTAL SUSPENDED SOLIDS**

TSS	< 5	mg/L	5	1	SM2540D	11/12/2013 12:27	DSBAKE1
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Site: BioReactor 2 Inf

Collection Date: 06-Nov-13 8:17 AM

**Sample #: 2013027422**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)</u></b>								
Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	187	mg/L		0.5	10	EPA 200.7	11/13/2013 12:34	MHH7131
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Nickel (Ni)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Selenium (Se)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Thallium (Tl)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Vanadium (V)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:00	DJSULL1

# Certificate of Laboratory Analysis

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*This report shall not be reproduced, except in full.***Order # J13110158**

Site: BioReactor 2 Inf

Collection Date: 06-Nov-13 8:17 AM

**Sample #: 2013027422**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
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Site: BioReactor 2 Eff

Collection Date: 06-Nov-13 8:17 AM

**Sample #: 2013027423**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
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**NITRITE + NITRATE (COLORIMETRIC)**

Nitrite + Nitrate (Colorimetric)	< 0.01	mg-N/L		0.01	1	EPA 353.2	11/15/2013 11:34	BGN9034
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**INORGANIC IONS BY IC**

Bromide	86	mg/L		5	50	EPA 300.0	11/11/2013 16:01	JAHERMA
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Chloride	6300	mg/L		100	1000	EPA 300.0	11/11/2013 16:01	JAHERMA
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**Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)**

Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
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**TOTAL RECOVERABLE METALS BY ICP**

Boron (B)	194	mg/L		0.5	10	EPA 200.7	11/13/2013 12:38	MHH7131
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**TOTAL RECOVERABLE METALS BY ICP-MS**

Arsenic (As)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Cadmium (Cd)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Chromium (Cr)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Copper (Cu)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Nickel (Ni)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Selenium (Se)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Silver (Ag)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Thallium (Tl)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Vanadium (V)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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Zinc (Zn)	< 5	ug/L		5	5	EPA 200.8	11/18/2013 15:13	DJSULL1
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**SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)**

Vendor Parameter	Complete					Vendor Method		V_AS&C
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Site: Bio Pilot Eff

Collection Date: 06-Nov-13 2:15 PM

**Sample #: 2013027424**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
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**NITRITE + NITRATE (COLORIMETRIC)**

Nitrite + Nitrate (Colorimetric)	< 0.01	mg-N/L		0.01	1	EPA 353.2	11/15/2013 11:29	BGN9034
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**Mercury by EPA 200.8 - (Analysis Performed by Applied Speciation and Consulting, LLC)**

Vendor Parameter	Complete	ug/l				Vendor Method		V_AS&C
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# Certificate of Laboratory Analysis

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Site: Bio Pilot Eff

Collection Date: 06-Nov-13 2:15 PM

**Sample #: 2013027424**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	186	mg/L		0.5	10	EPA 200.7	11/13/2013 12:42	MHH7131
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	72.7	ug/L		10	10	EPA 200.8	11/20/2013 14:12	DJSULL1
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Cadmium (Cd)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Chromium (Cr)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Copper (Cu)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Nickel (Ni)	19.0	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Selenium (Se)	63.5	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Silver (Ag)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Thallium (Tl)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Vanadium (V)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1
Zinc (Zn)	< 10	ug/L		10	10	EPA 200.8	11/18/2013 15:16	DJSULL1

**SELENIUM SPECIATION - (Analysis Performed by Applied Speciation and Consulting, LLC)**

Vendor Parameter	Complete	Vendor Method	V_AS&C
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Site: FILTER BLANK

Collection Date:

**Sample #: 2013027425**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>DISSOLVED METALS BY ICP-MS</u></b>								
Selenium (Se)	< 1	ug/L		1	1	EPA 200.8	11/20/2013 13:26	DJSULL1

Site: METALS TRIP BLANK

Collection Date: 28-Oct-13 4:35 PM

**Sample #: 2013027426**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>TOTAL RECOVERABLE METALS BY ICP</u></b>								
Boron (B)	< 0.05	mg/L		0.05	1	EPA 200.7	11/13/2013 11:41	MHH7131



# Certificate of Laboratory Analysis

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**Order # J13110158**

Site: METALS TRIP BLANK

Collection Date: 28-Oct-13 4:35 PM

**Sample #: 2013027426**

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	DF	Method	Analysis Date/Time	Analyst
<b><u>TOTAL RECOVERABLE METALS BY ICP-MS</u></b>								
Arsenic (As)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Cadmium (Cd)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Chromium (Cr)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Copper (Cu)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Nickel (Ni)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Selenium (Se)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Silver (Ag)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Thallium (Tl)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Vanadium (V)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1
Zinc (Zn)	< 1	ug/L		1	1	EPA 200.8	11/18/2013 14:09	DJSULL1



**APPLIED SPECIATION  
AND CONSULTING, LLC**

18804 Northcreek Parkway Bothell, WA, 98011  
Tel: (425) 483-3300 Fax: (425) 483-9818  
[www.appliedspeciation.com](http://www.appliedspeciation.com)

November 27, 2013

Jay Perkins  
Duke Energy Analytical Laboratory  
Mail Code MGO3A2 (Building 7405)  
13339 Hagers Ferry Rd.  
Huntersville, NC 28078  
(704) 875-5245

Project: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling) (LIMS# J13110158)

Mr. Perkins,

Attached is the report associated with five (5) aqueous samples submitted for total mercury, hexavalent chromium, and selenium speciation analyses on November 11, 2013. The samples were received in a sealed cooler at -0.2°C on November 12, 2013. Mercury quantitation was performed via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS). Hexavalent chromium analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Selenium speciation analysis was performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). Any issues associated with the analyses are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

A handwritten signature in black ink, appearing to read "Jeremy Maute".

Jeremy Maute  
Project Coordinator  
Applied Speciation and Consulting, LLC

Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins  
Duke Energy Analytical Laboratory  
Mail Code MGO3A2 (Building 7405)  
13339 Hagers Ferry Rd.  
Huntersville, NC 28078

Project: Frontier Pilot (Belews Creek) WWTS (Weekly Sampling) (LIMS# J13110158)

November 27, 2013

## 1. Sample Reception

Four (4) aqueous samples were submitted for hexavalent chromium and selenium speciation analyses on November 11, 2013. Four (4) additional samples were submitted for total mercury quantitation. All samples were received in acceptable condition on November 12, 2013 in a sealed container at -0.2°C.

All samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and were designated discrete sample identifiers. The 40mL borosilicate glass vials submitted for total mercury were preserved with bromine monochloride (BrCl) solution. The resulting samples were stored in a secure polyethylene container, known to be free from trace metals contamination, until the analyses could be performed.

An aliquot of each sample submitted for hexavalent chromium analysis was filtered (0.45µm) and stored in a secure refrigerator maintained at a temperature of 4°C, until the analyses could be performed.

An aliquot of each sample requiring selenium speciation evaluation was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS).

## 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

Total Mercury Quantitation by CV-ICP-MS All samples and preparation blanks for total mercury quantitation were preserved with 2% (v/v) BrCl. The resulting samples were analyzed for mercury via cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS).

Hexavalent Chromium Analysis by IC-ICP-DRC-MS Prior to analysis, all samples were injected directly into autosampler vials. No further sample preparation was performed as any chemical alteration of the samples may shift the equilibrium of the system resulting in changes in speciation ratios.

Selenium Speciation Analysis by IC-ICP-CRC-MS Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45 $\mu$ m) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimum interval of every ten analytical runs.

Total Mercury Quantitation by CV-ICP-MS The sample fractions for total mercury quantitation were analyzed by cold vapor inductively coupled plasma mass spectrometry (CV-ICP-MS) on November 18, 2013. Aliquots of each sample are reacted with a reductant in-line and transported to a gas-liquid separator. The volatile elemental mercury that is formed is then swept by a stream of argon gas into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and separated on the basis of their mass-to-charge ratio ( $m/z$ ) by a mass spectrometer. A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Hexavalent Chromium Analysis by IC-ICP-DRC-MS Each sample for hexavalent chromium analysis was analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on November 26, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7)

gradient. The eluting chromium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios ( $m/z$ ). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

*Selenium Speciation Analysis by IC-ICP-CRC-MS* Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS) on November 15, 2013. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic ( $\text{pH} > 7$ ) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (CRC) containing a reaction gas which preferentially reacts with interfering ions of the same target mass to charge ratios ( $m/z$ ). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

#### **4. Analytical Issues**

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with these samples were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL values for methylseleninic acid and selenomethionine are calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

The eMDL values for hexavalent chromium and mercury are calculated using the standard deviation of the preparation blanks preserved and analyzed concurrently with the submitted samples.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

A handwritten signature in black ink, appearing to read 'J. Maute', with a stylized flourish extending to the right.

Jeremy Maute  
Project Coordinator  
Applied Speciation and Consulting, LLC

## Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy

Project Name: Frontier Pilot (Belews Creek) WWTS (Weekly Sampling)

Contact: Jay Perkins

LIMS #J13110158

Date: November 27, 2013

Report Generated by: Jeremy Maute

Applied Speciation and Consulting, LLC

**Sample Results**

Sample ID	Total Hg	Cr(VI)	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Unknown Se Species (n)
FGD Purge Eff	NR	1.8	74.0	50.5	ND (< 2.1)	3.1	ND (< 1.7)	0 (0)
BioReactor 1 Inf	0.232	ND (< 1.0)	18.5	35.9	ND (< 0.53)	0.65	ND (< 0.43)	2.64 (1)
BioReactor 2 Inf	0.0345	NR	NR	NR	NR	NR	NR	NR
BioReactor 2 Eff	0.0076	1.6	ND (< 0.47)	ND (< 0.30)	ND (< 0.53)	ND (< 0.43)	ND (< 0.43)	0 (0)
Bio Pilot Eff	0.0725	ND (< 1.0)	17.3	36.3	ND (< 0.53)	ND (< 0.43)	ND (< 0.43)	0 (0)

All results reflect the applied dilution and are reported in µg/L

NR = Analysis not requested

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

## Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy

Project Name: Fontier Pilot (Belews Creek) WWTS (Weekly Sampling)

Contact: Jay Perkins

LIMS #J13110158

Date: November 27, 2013

Report Generated by: Jeremy Maute

Applied Speciation and Consulting, LLC

**Quality Control Summary - Preparation Blank Summary**

Analyte (µg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 5x	eMDL 250x	eMDL 1000x
Hg	-0.0003	-0.0002	-0.0007	-0.0004	-0.0004	0.0002	0.0001	0.0006	-	-
Cr(VI)	-0.8	-0.4	-0.2	0.0	-0.3	0.3	0.002	-	-	1.0
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.47	1.9
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.001	-	0.30	1.2
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.53	2.1
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.43	1.7
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.002	-	0.43	1.7

eMDL = Estimated Method Detection Limit

\*Please see narrative regarding eMDL calculations

**Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Hg	NIST 1641d	1568	1626	103.7
Cr(VI)	LCS	1.000	1.054	105.4
Se(IV)	LCS	9.57	9.28	96.9
Se(VI)	LCS	9.48	9.22	97.3
SeCN	LCS	8.92	8.55	95.9
MeSe(IV)	LCS	6.47	6.23	96.3
SeMe	LCS	9.32	8.84	94.8



## Total Mercury, Hexavalent Chromium, and Selenium Speciation Results for Duke Energy

Project Name: Frontier Pilot (Belews Creek) WWTS (Weekly Sampling)

Contact: Jay Perkins

LIMS #J13110158

Date: November 27, 2013

Report Generated by: Jeremy Maute

Applied Speciation and Consulting, LLC

**Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Hg	Batch QC	0.1934	0.1955	0.1945	1.1
Cr(VI)	Bio Pilot Eff	ND (< 1.0)	ND (< 1.0)	NC	NC
Se(IV)	Bio Pilot Eff	17.33	18.07	17.70	4.2
Se(VI)	Bio Pilot Eff	36.27	38.29	37.28	5.4
SeCN	Bio Pilot Eff	ND (< 0.53)	ND (< 0.53)	NC	NC
MeSe(IV)	Bio Pilot Eff	ND (< 0.43)	ND (< 0.43)	NC	NC
SeMe	Bio Pilot Eff	ND (< 0.43)	ND (< 0.43)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

**Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Hg	Batch QC	2.000	2.347	107.6	2.000	2.320	106.3	1.2
Cr(VI)	Bio Pilot Eff	1000	986.4	98.6	1000	986.9	98.7	0.0
Se(IV)	Bio Pilot Eff	1390	1312	93.1	1390	1344	95.4	2.4
Se(VI)	Bio Pilot Eff	1261	1203	92.4	1261	1224	94.1	1.7
SeCN	Bio Pilot Eff	1144	1037	90.6	1144	1056	92.3	1.8





# CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM

**Duke Energy**  
**Duke Energy Analytical Laboratory**  
 Mail Code MGO3A2 (Building 7405)  
 13339 Hagers Ferry Rd  
 Huntersville, N. C. 28078  
 (704) 875-5245  
 Fax: (704) 875-4349

**Analytical Laboratory Use Only**

ORDER# 58 MATRIX: OTHER  
713 110132 11/8

Logged By B. G. ... Date & Time 11/7/13 11:00

SAMPLE PROGRAM Ground Water NPDES  
 Drinking Water UST  
 RCRA Waste RCRA Waste

AS&C  
 PO #650910

Cooler Temp (C) 0.3

Preserv.: 1=HCL  
 2=H<sub>2</sub>SO<sub>4</sub> 3=HNO<sub>3</sub>  
 4=Ice 5=None

Page 19 of 19  
 Page 1 of 2  
**DISTRIBUTION**  
 ORIGINAL to LAB,  
 COPY to CLIENT

1) Project Name **Frontier Pilot (Belews Creek) WWTS (Weekly Sampling)**

2) Client: **Bill Kennedy, Wayne Chapman**

5) Business Unit: **20006** 6) Process: **SFHDW1205** Mail Code:

8) Oper. Unit: **FHGO** 9) Res. Type: **3500** 10) Reso. Center: **1830000**

Customer to complete all appropriate non-shaded areas.

**LAB USE ONLY**

11) Lab ID

2013027312  
2013027314  
2013027315  
2013027316  
2013027317  
2013027318  
2013027319  
2013027320  
2013027321  
2013027322

Se Speciation Bottle ID	13 Sample Description or ID	Sampling conducted: Every Wednesday			17 Comp.	18 Grab	TDS, TSS	Br, Cl (Dionex)	Metals* + Hg**	Se (IMS), filtered	Hg 200.8 (V, AS&C)	Nitrate	Cr, Se, speciation - vendor to AS&C (Important to place filled bottle back into both baggies)
		Date	Time	Signature									
419	FGD Purge Eff	11-6-13	0805	TO			1	1	1	1			1
420	EQ Tank Eff.	11-6-13	0810	TO					1	1			
421	BioReactor 1 Inf	11-6-13	0815	TO			1	1	1**	1	1	1	1
422	BioReactor 2 Inf	11-6-13	0817	TO					1**		1		
423	BioReactor 2 Eff	11-6-13	08:17	TO				1	1**		1	1	1
424	Bio Pilot Eff	11-6-13	14:15	RE					1**	1	1	1	1
	mF Pilot Eff	N/A	N/A	NO sample			1	1	1**	1	1	1	1
425	Filter Blk									1			
426	Metals Trip Blk	11/28/13	1635	D. Bal					1**				

Filtering of the Se is performed in the field please provide a filter blank too.

Customer to sign & date below - fill out from left to right.

1) Relinquished By Phil Gossitt Date/Time 11/6/13 14:30

2) Accepted By [Signature] Date/Time 11/7/13 11:00

3) Relinquished By [Signature] Date/Time 11/11/13 8:00

4) Accepted By [Signature] Date/Time 11/11/13 8:00

5) Relinquished By [Signature] Date/Time 11/11/13 8:00

6) Accepted By [Signature] Date/Time 11/11/13 8:00

7) Relinquished By [Signature] Date/Time 11/11/13 8:00

8) Accepted By [Signature] Date/Time 11/11/13 8:00

9) Seal/Locked By [Signature] Date/Time 11/11/13 8:00

10) Seal/Locked Opened By [Signature] Date/Time 11/11/13 8:00

11) Seal/Locked By [Signature] Date/Time 11/11/13 8:00

12) Seal/Locked Opened By [Signature] Date/Time 11/11/13 8:00

Comments

Customer, IMPORTANT!  
 Please indicate desired turnaround.

22) Requested Turnaround

14 Days \_\_\_\_\_

\*7 Days \_\_\_\_\_

\*48 Hr \_\_\_\_\_

\*Other \_\_\_\_\_

\* Add. Cost Will Apply

\* B by TRM/ICP As, Cd, Cr, Cu, Ni, Se, Ag, Zn, V, TI by TRM/IMS 1\*\*=No Hg

B6  
 11/8

BIO & INF mis labeled should  
 HADG 133N BIO2 EFF